Improving mechanical strength of YBCO bulk superconductors by addition of Ag

J. V. J. Congreve, Y. H. Shi, K. Y. Huang, A. R. Dennis, J. H. Durrell, and D. A. Cardwell

Abstract— **The widespread use of ceramic (RE)$Ba_2Cu_3O_7$ bulk superconductors (RE-123), where RE=Y, Gd or Sm, is generally hindered by their poor mechanical properties. While a large number of techniques can be used to improve the mechanical properties of conventional ceramic materials, many of these are incompatible with the growth of single grain, bulk RE-123 superconductors using the top seeded melt growth (TSMG) process. Complications arise due to the need to minimize the degradation of the superconducting properties and produce a single-grained sample. Nonetheless, the addition of Ag to RE-Ba-Cu-O [(RE)BCO] precursor powder has been demonstrated to be effective in improving the mechanical properties of single grain bulk superconductors fabricated by TSMG without deleterious effects on performance. Although large single grains of $GdBa_2Cu_3O_7$-Ag and $SmBa_2Cu_3O_7$-Ag have been successfully and reliably grown, it has proven more difficult to grow large single grains of $YBa_2Cu_3O_7$-Ag. We recently demonstrated the growth of single grain $YBa_2Cu_3O_7$-Ag bulk superconductors that exhibit relatively good superconducting properties. In this work, we report the flexural stress at a number of locations within $YBa_2Cu_3O_7$ single grains grown by TSMG with and without additional liquid phase and with silver addition. In addition, we have compared the distribution of the failure stress with the pore and silver distribution.**

***Index Terms*—Mechanical properties, Superconducting materials, TSMG, YBCO, YBCO-Ag**

I. Introduction

SINGLE grain (RE)$Ba_2Cu_3O_7$ [(RE)BCO] bulk high temperature superconductors (HTS), where RE=Y, Gd or Sm, are able to trap significantly larger magnetic fields than those generated by conventional permanent magnets [1],[2]. This enables a wide range of potential practical applications for these materials, including Maglev trains, energy storage systems and trapped flux devices [3]-[5]. However, significant stresses are generated during the magnetization and operation of these technologically important materials within the bulk sample due to the large Lorentz forces. The brittle nature of these materials limit significantly the mechanical properties and hence the exploitation of superconducting properties. This, in turn, reduces the suitability of these materials for applications involving relatively high magnetic fields [6]-[7].

Although the mechanical properties are ultimately the limitation on the achievable superconducting properties, little research has been performed on measuring the mechanical properties or on the magnitude of the stresses generated during the magnetisation process. Bulk (RE)BCO single grains tend to fracture when the internal stresses exceed 10- 30 MPa [8].

The presence of poorly connected grain boundaries in single grain (RE)BCO superconductors reduces significantly the flow of supercurrent within the bulk sample, which, in turn, reduces significantly the size of the current loop and therefore the magnitude of the trapped field generated by the sample [9], [10]. This means that these materials are only useful for practical applications when processed in the form of large, single grains [11]-[13]. The top seeded melt growth (TSMG) process is one of the most common and simplest processes used widely to fabricate large single grains of (RE)BCO [9],[14].

Although the TSMG process is one of the simplest fabrication techniques available for the processing of individual single grains, it requires the optimization of many interdependent process parameters. Alloying elements can have a significant impact on these parameters and make successful TSMG growth difficult. For example: the addition of Ag alters the peritectic decomposition temperature by 30 °C. Due, in part, to these considerations it is only recently that large single grains of YBCO-Ag have been grown reliably and successfully [15],[16].

Several authors have reported that the addition of silver to (RE)BCO is beneficial to improving its mechanical properties [17],[18] and to increase significantly the fracture toughness and bending strength [18]-[20], without degrading the superconducting properties, unlike many other alloying elements [17],[21],[22]. Other studies have shown that the inclusion of silver is able to enhance the critical current density significantly in bulk superconductors [21]. The increase in fracture toughness is thought to be due to the reduction in porosity and cracking [21],[23] that occurs when silver is present. A number of these studies have suggested that pores in the single grain microstructure are filled by silver in the melt [17],[24]. Many studies have also suggested that the Ag precipitates are able to modify the distribution of residual stresses [25], which are the leading factors in the propagation of dislocations and cracks [21].

The incorporation of the silver within the Y-123 matrix is important when this dopant is used to improve the mechanical properties of (RE)BCO [26]. It is thought that the Ag additions within the RE-123 phase matrix modify the residual stress distribution within the bulk sample [21]. Strains are generated during cooling due to the differing thermal expansion coeffi-

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cients of Y-123 and Ag [27]. It has also been found that large stresses are created around Ag and Y-211 inclusions after processing [25]. Ceramic materials subjected to compressive stress is less likely to crack or fail than under tension, so there is potential to engineer the sample microstructure via the addition of Ag to optimize the intrinsic resilience to such forces.

The successful and reliable growth of YBCO-Ag by liquid-phase-enriched TSMG [15],[16] has enabled the mechanical and superconducting properties of YBCO-Ag to be investigated in detail. The variation in the tensile strength has been investigated along the *c*-axis within the bulk single grain. The distribution of tensile strength has been compared with the distribution of pores and silver. The tensile strength at a number of locations has also been compared for YBCO grown by standard TSMG, and YBCO and YBCO-Ag grown by liquid-phase-enriched TSMG.

II. Method

A. Sample growth

Three samples of YBCO were prepared by conventional TSMG [28]. 46 g grams of precursor powder mixed from 99.9 % purity powders of Y-123:Y-211:CeO_2 in a mass ratio 150:50:1 was pressed uniaxially in a cylindrical die of diameter 30 mm.

Three further samples of YBCO were grown by liquid-phase enriched TSMG [28], labelled LR YBCO. Precursor powder was mixed from 99.9 % purity powders of Y-123:Y-211:CeO_2 in a mass ratio of 150:50:1. Liquid-phase-rich powder was mixed from Yb_2O_3:$Ba_3Cu_5O_8$:BaO_2 in the ratio 5.0:5.6:1.0 and calcined once at 850 °C for 5 h. Composite green pellets were pressed with a 4.6 g layer of liquid-phase-rich powder below 46 g of precursor powder.

Finally, three samples of YBCO-Ag were grown by liquid-phase-enriched TSMG [16]. Precursor powder was mixed from 99.9 % purity powders of Y-123:Y-211:CeO_2:Ag_2O in a mass ratio of 150:50:1:20. Liquid-phase-rich powder was mixed and calcined as described above. Composite green pellets were pressed with a 4.6 g layer of liquid-phase powder below 46 g of precursor powder.

Following growth the samples were annealed in oxygen to transform the tetragonal structure to the superconducting orthorhombic structure and to optimise T_c [29].

B. Measurement of mechanical properties

Each sample was cut in half to expose a rectangular cross section. One half of each of sample was then cut further into flexural beams of approximate size 2.0 mm x 1.5 mm x 20.0 mm, and labelled as shown in Fig. 1, using a diamond cutting wheel. Each beam was cleaned in warm acetone to remove any wax remnants from the cutting process and then polished lightly with grade 2400 silicon carbide paper to ensure each edge was smooth.

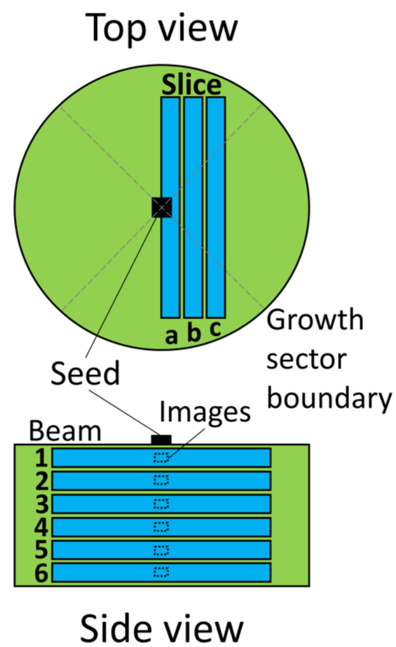

Fig. 1. A schematic of the locations of the beams used for mechanical testing.

A three-point bend test was used to measure the flexural strength at room temperature. The testing referred to ASTM C1161. A load was applied in the *c*-axis direction at a speed of 0.15 mm/min using an electro-mechanical tensile testing machine. The lower surface of each beam was always in tension in this arrangement. This enabled the flexural strength along the *ab*-axes to be determined. The experimental arrangement is shown in Fig. 2. The beams were tested to failure and the following equation was used to calculate the flexural stress at failure:

$$\sigma_f = \frac{3Fl}{2bd^2} \tag{1}$$

where F is the load at failure, l is the support span, b is the width of test beam (approximately 1.5 mm) and d is the depth of the test beam (approximately 2.0 mm).

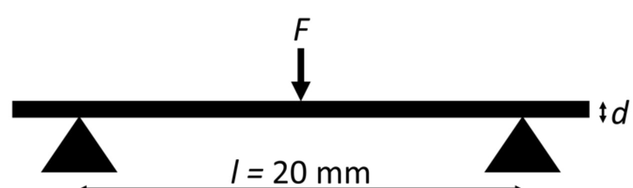

Fig. 2. Schematic of the three-point bend test setup.

C. Microstructure and composition analysis

The remaining half of each sample was polished and imaged using an optical microscope at 50 x magnification to observe the pore and silver distribution for the relevant samples. Images were taken at the centre of each of the flexural beams from slice a. ImageJ software [30] was used to analyse each of the images to quantify the area fraction occupied by pores and, where relevant, silver agglomerates. In addition the average size of the pores and silver agglomerates in each image was recorded. The colour threshold was adjusted to highlight the pores or silver agglomerates in an image then the analyse particles tool was used to collect data on the size and area of the image occupied by these pores or agglomerates.

III. Results

A. Mechanical properties

Three-point bend tests were undertaken on 41 beams from each of YBCO and LR YBCO and 47 beams of YBCO-Ag. 7 beams from the YBCO-Ag samples and 13 beams from each of the LR YBCO and YBCO bulks, had large cracks or pores present which prevented their use in the three-point bend test. The majority of these beams were from near the sample centre, which is as expected given that more pores are usually present at the centre of the sample than at the edge.

The average stress at failure for each flexural beam location in each of the slices is shown in Fig. 3. The beams that were unable to be tested have been excluded from the average value, but have been recorded as having zero strength for the purpose of representation in the error bars.

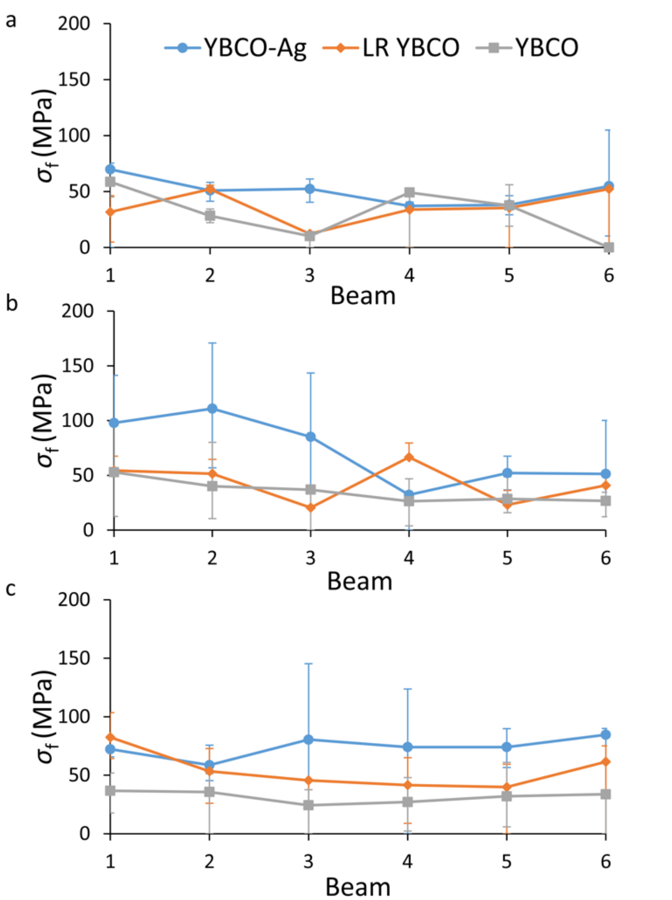

Fig. 3. The average flexural strength of each beam in a) slice a, b) slice b, and c) slice c.

YBCO-Ag beams had the highest average mechanical strength for the majority of the flexural tests. Only three corresponding flexural beams had noticeably higher strength than the YBCO-Ag beams. In addition, the highest overall maximum mechanical strength for each corresponding beam was recorded in the YBCO-Ag for every location measured except four, as shown by the error bars in Fig. 3. A maximum mechanical strength of 170 MPa was seen in the YBCO-Ag beams. However, there was significant fluctuation in the mechanical strength for corresponding beams from different samples of the same type. This large fluctuation in mechanical strength is typical of brittle ceramics.

The provision of additional liquid during growth also improves the mechanical strength, although much less so than the addition of Ag.

Although the overall mechanical properties of the system are likely to be limited by the regions with the worst mechanical properties, and in this case a number of locations in the YBCO-Ag samples where the flexural strength was low did not have a greater flexural strength than the strongest regions in the YBCO samples without Ag, we have shown that there is a significant improvement in the overall average mechanical properties when silver is added. This study has also highlighted the complexity involved in improving the mechanical strength, alongside the variability in the mechanical properties within each single grain. Although the mechanical properties are not better in every location within the YBCO-Ag samples than in the YBCO samples, this work has indicated that with further optimisation we will be able to achieve better mechanical properties throughout the YBCO-Ag samples, thus with further work we can eliminate the ‘bottle-neck’ in the fracture of these brittle samples.

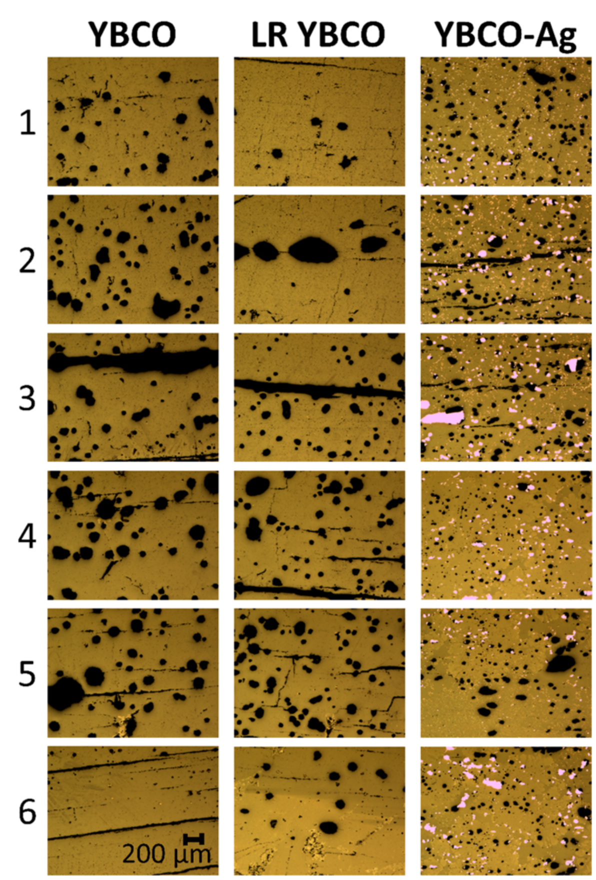

Fig. 4. Microscope images corresponding approximately to the centre of each beam located in slice 1.

B. Microstructure and composition analysis

The microstructure was imaged at locations corresponding to the centre of each beam from slice a (a representative example from one of each type of sample is shown in Fig. 4). The microstructure of samples of the same type (either YBCO, LR YBCO or YBCO-Ag) show very similar trends in the image taken from each corresponding bar. Although the microstructures of YBCO and LR YBCO are very similar, the microstructure of YBCO-Ag is significantly different. The pores (shown as black circular regions, with cracks as thin black regions) in YBCO-Ag are significantly smaller. There are also a large number of very small silver agglomerates accompanying some larger silver agglomerates (shown as bright yellow, almost white in colour) which appear to have filled areas that would otherwise have been pores.

The area of each image occupied by pores and, where relevant, silver, as determined by ImageJ software, averaged over all three samples is shown in Fig. 5, alongside a plot of the average size of the pores and silver in each sample. The average porosity is much lower in the YBCO-Ag samples than in either the YBCO or LR YBCO samples at all locations except locations 3 and 6, therefore some of the pores must have been filled by silver agglomerated in the YBCO-Ag samples. The average area occupied by pores in the LR YBCO is slightly lower than that of the standard YBCO sample, but not significantly lower.

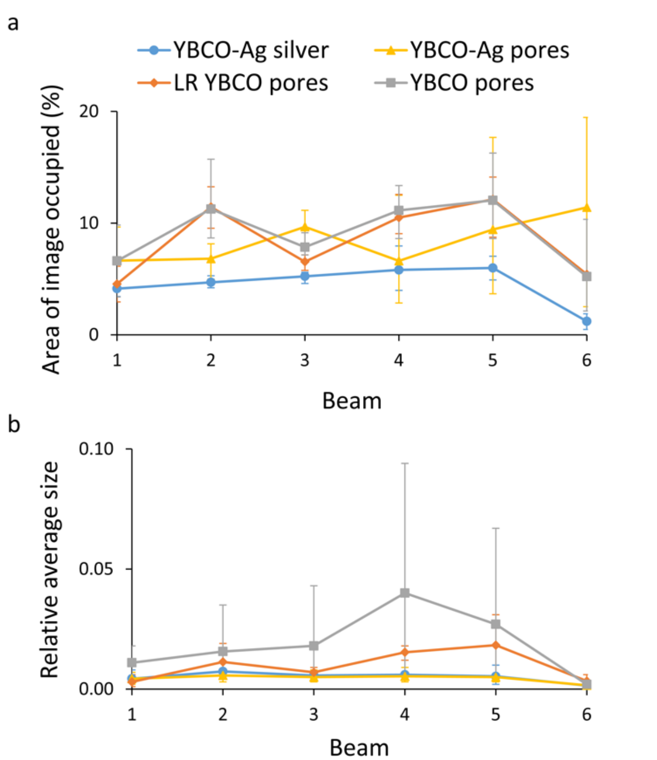

Fig. 5. The average variation in porosity between the beams considered in terms of a) the average area occupied by pores, and b) the average size of the pores present at each location.

The average size of the pores is significantly different in each of the samples. The average pore size in each image of the standard YBCO samples is much larger than that of all of the other samples and shows a much greater variation between subsequent beam locations. In addition, the error bars show a significant variation in the average pore size at each location between the three samples of standard YBCO. The average pore size is significantly lower in the YBCO-Ag samples than in the LR YBCO samples. The average pore size in the YBCO-Ag sample is also very consistent between beam locations and between the three samples of YBCO-Ag.

C. Relation between microstructure and composition analysis

It is accepted widely that porosity in the microstructure of YBCO bulk superconductors has a negative impact on the mechanical properties of the sample, both in terms of the total area occupied by pores and the size of the pores present [31].

The addition of silver reduces both the area fraction occupied by pores and the average size of the pores, as shown in Fig. 5. Both the area fraction occupied by pores and the average pore size have been plotted against the flexural strength for beams from slice a in Fig. 6. Studying each system separately indicates that there is a slight negative correlation between the average pore size and the flexural strength and the area occupied by pores and the flexural strength. The larger the area occupied by pores and the larger the average pore size, the lower the flexural strength. Therefore, the reduction in both area occupied by pores and size of the pores caused by their partial filling with silver would appear to be, at least in part, responsible for the increase in local strength associated with the addition of silver. Due to the nature of failure of ceramic materials, there is a large variation in the mechanical strength of the same material, even for sub-specimens taken from the same original piece of material, and this is likely to be responsible for the large variation in flexural stress and may be why only a slight negative correlation was observed.

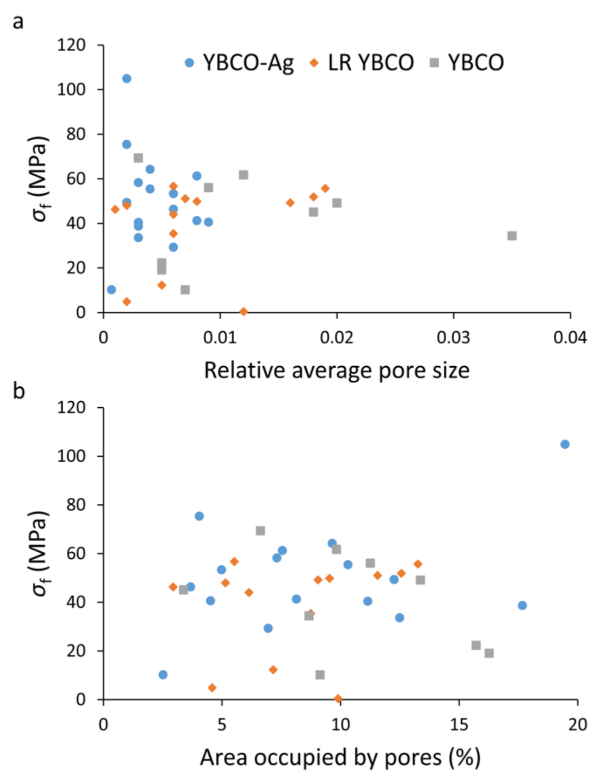

Fig. 6. The relationship between flexural strength and porosity related to a) the average size of the pores, b) the area occupied by pores.

In the LR YBCO samples, the reduction in area occupied by pores and the reduction in the average pore size is, at least in part responsible for the higher local flexural strength observed in the majority of locations in comparison to standard YBCO. Alongside filling some of the pores, the provision of additional liquid-phase during processing may also enable small, sharp cracks to be filled or blunted, hence providing additional mechanisms to improve the mechanical strength of the material. The addition of Ag to the samples may have a similar effect, filling the sharp microcracks, and hence blunting the sites at which crack initiation and propagation is most and first likely to occur.

IV. Conclusion

The provision of additional liquid during the growth process improves the mechanical strength of YBCO bulk superconductors and the addition of Ag improves the local mechanical strength significantly. Small beams of YBCO-Ag were able to withstand stresses over 170 MPa before failure. This increase in failure stress is due to a reduction in the area occupied by pores and a reduction in the average pore size. The added silver partially filled most of the pores, hence the area occupied by and average size of the pores was significantly reduced.

The negative correlation between the pore size and area occupied by pores was limited due to the nature of the variation in failure stress of ceramic materials and the inherent variation between YBCO samples grown by TSMG.

This observed significant improvement in the mechanical properties due to the addition of silver to the YBCO system will significantly enhance the viability of this material for use in practical applications.

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Additional data related to this publication is available at the University of Cambridge data repository [https://doi.org/10.17863/CAM.31455]. All other data accompanying this publication are directly available within the publication.

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